SPECTROGRAPHIC ANALYSIS

I. ESTIMATION OF BORON IN BIOLOGICAL MATERIALS

II. QUANTITATIVE ANALYSIS OF SULFANILAMIDE-SULFATHIAZOLE MIXTURES

BY

DOUGLAS ARVID SKOOG

B.S., Oregon State College, 1940

AN ABSTRACT OF A THESIS

SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF DOCTOR OF PHILOSOPHY IN CHEMISTRY IN THE GRADUATE SCHOOL OF THE UNIVERSITY OF ILLINOIS, 1943

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Spectrographic Analysis
I. Estimation of Boron in Biological Materials

Introduction

In recent years it has become apparent that boron is an essential plant nutrient. In order to study its function in plants, an analytical method is required which is capable of estimating boron concentrations from 1 to 120 parts per million. Usually it is desirable that such a method shall not require a large sample weight. Studies on the use of radioactive boron in cancer therapy have also necessitated a boron method of similar sensitivity. These needs gave rise to this present study of the application of a spectrochemical method to the estimation of boron in biological materials.

The high potential, condensed spark was chosen as a source for this work because of the higher degree of precision obtainable with this type of excitation. Because there is only slight heating of the electrodes, the spark excitation is particularly adaptable to solution samples.[1] Kiers and Englis have described a very simple and satisfactory method for the continuous introduction of solutions into the spark.[3] Their method was adopted for handling the liquid extract from the ash of the organic materials containing boron.

Experimental

In the preliminary work an attempt was made to apply a wet oxidation procedure for the preparation of samples for analysis. This was not successful because boron was lost by volatilization from the hot acid solutions used. However, when samples were mixed with sodium carbonate and dry ashed in platinum crucibles, a complete recovery of boron was obtained.

A Bausch and Lomb Littrow Spectrograph was employed for this work. The slit width was set at 0.06 mm. Plate den-

sities were determined with a Leeds and Northrup recording microphotometer. Eastman type II-0, ultraviolet sensitized plates were used. Plates were calibrated with a stepped sector using an iron spark as a source. This means of plate calibration was compared with a method involving the variation of exposure time of a source of constant intensity. The two methods gave similar plate calibration curves. Also, the effect of a small background adjacent to the line used for plate calibration with the stepped sector was studied and found to be insignificant.

The boron line at 2497.7 Angstrom units was chosen for the analytical work. Gold was found to be satisfactory as an internal standard element, and its line at 2675.9 Angstrom units was used. The logarithm of the intensity ratio of these two lines was found to have a linear relation to the logarithm of boron concentration.

It was found necessary to use basic solutions when sparking the samples in order to avoid a loss in boron due to the heat developed in the electrode holder as a series of solutions was being excited.

The effect of extraneous ions on the gold and boron line densities was studied. It was found that the hydroxyl ion caused the boron line density to increase and reach a maximum at a ratio of four moles of sodium hydroxide to one of boric acid. Higher concentrations of hydroxyl ion had no effect on the ratios of the line densities. Sodium and potassium ions caused both the boron and gold line densities to be decreased. However, the ratio of the line densities was not affected by these ions. Phosphate ions had no effect upon the intensity relationship of the chosen pair of lines. A slight initial interference of a phosphorus line near the selected line of boron was eliminated by reduction of the slit width to 0.06 mm. or less under which conditions resolution was possible.

A series of synthetic samples was analysed by this pocedure. The average error for twelve samples was found to be 5.5% of the total amount of boron present. The boron

content of several types of plant samples was estimated with a precision of 5%. Measured amounts of boric acid were added to a few of these plant samples, and the recovery of boron was measured. The average error for seven samples treated in this manner was 4%. Thus, the average error for the analysis is considered to be about 5%. The limit of sensitivity is 0.005 mg. boron.

II. Quantitative Analysis of Sulfanilamide-Sulfathiazole Mixtures

Introduction

The use of the sulfa drugs in chemical therapy has made the quantitative analysis of mixtures of these drugs of considerable importance. Mixtures of sulfanilamide and sulfathiazole have found use in dessings for wounds, and a means of analysis of this particular mixture was desirable.

Although some work has been reported on the determination of the total sulfa drug content of mixtures,[2,4] little has been done towards estimating the concentration of individual drugs present in mixtures. After a consideration of the structure of sulfanilamide and sulfathiazole, it was thought the ultraviolet absorption spectra of the two compounds should be considerably different and that this might be used as a basis for the quantitative estimation of the two drugs.

Experimental

A Bausch and Lomb Medium Quartz Spectrograph was employed in this work. The slit was set at a width of 0.06 mm. A Wood's type of hydrogen discharge tube was used to give a continuous spectrum of constant intensity. The spectra were recorded on Eastman polychrome plates. Each plate was calibrated by making a series of separate exposures of different lengths of time. A one cm. cell with quartz windows was used to contain the liquids during the exposures. Extinction values were calculated from the densities of the solvent and

solution spectra.

The ultraviolet absorption curves for ethyl alcohol solutions of sulfanilamide and sulfathiazole were prepared. The sulfanilamide gave a single absorption peak at 2610 Angstrom units while the sulfathiazole had two peaks, one at 2580 Angstrom units and the other at 2875 Angstrom units.

Both drugs were found to obey Beer's Law at both 2600 and 2875 Angstrom units. By assuming that the two drugs acted independently of one another in solution, it was possible to calculate the amount of each drug present from the extinction coefficients of the mixture at 2600 and 2875 Angstrom units.

The procedure was found to be sensitive to one milligram of each drug per liter of solution. A series of sixteen mixtures was examined in this manner and the amount of each drug was estimated. The average error for the sulfanilamide determination was found to be four percent while the error for the sulfathiazole estimation was six per cent.

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Vita

The author was born in Willmar, Minnesota on May 4, 1918. He attended grammar school in Portland, Oregon until 1932 and graduated from Washburn High School in Minneapolis, Minnesota in 1936. His undergraduate work was done at Oregon State College from which he received his Bachelor of Science Degree in Chemistry in 1940. He began his work at the University of Illinois in the fall of the same year. He held a teaching assistantship in the Chemistry Department from 1940 until 1943.

He is a member of Sigma Xi, Phi Kappa Phi, Phi Lambda Upsilon, Pi Mu Epsilon and Alpha Chi Sigma.

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